



A Review of State-of-the-Art Separator Materials for Advanced Lithium-Based Batteries for Future Aerospace Missions

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Abstract

As NASA embarks on a renewed human presence in space, safe, human-rated, electrical energy storage and power generation technologies, which will be capable of demonstrating reliable performance in a variety of unique mission environments, will be required. To address the future performance and safety requirements for the energy storage technologies that will enhance and enable future NASA Constellation Program elements and other future aerospace missions, advanced rechargeable, lithium-ion battery technology development is being pursued with an emphasis on addressing performance technology gaps between state-of-the-art capabilities and critical future mission requirements. The material attributes and related performance of a lithium-ion cell's internal separator component are critical for achieving overall optimal performance, safety and reliability. This review provides an overview of the general types, material properties and the performance and safety characteristics of current separator materials employed in lithium-ion batteries, such as those materials that are being assessed and developed for future aerospace missions.

Introduction

As NASA embarks on a renewed human presence in space as part of the "U.S. Space Exploration Policy", we will require safe, human-rated, electrical energy storage and power generation technologies that will be able to demonstrate reliable performance in a variety of unique mission environments. NASA Constellation Program elements include the Crew Exploration Vehicle (Orion), crew and cargo launch vehicles (Ares I and Ares V, respectively), the Lunar Surface Access Module (Altair), extravehicular activity (EVA) surface suits and extraterrestrial rovers and habitats (Ref. 1). To address the future performance and safety requirements for the energy storage technologies that will enhance and enable these and other future aerospace missions, advanced rechargeable, lithium-ion battery technology development is being pursued with an emphasis on addressing performance technology gaps between state-of-the-art capabilities and critical future mission requirements.

Lithium-ion batteries have not been fully space-qualified for use as a main energy storage element for manned, human-rated aerospace missions. Currently, both cell and cell component development efforts are focused on improving the overall safety, as well as the electrochemical attributes, of such battery systems in order to optimize such for manned space applications. A key cell component of a lithium-ion battery which significantly impacts both of these performance features is the battery separator.

The function and reliability of the separator is critical for the optimal performance and safety of a lithium-ion cell. In nonaqueous, liquid-electrolyte cells that exemplify a lithium-ion cell chemistry, the separator is typically a nonelectrically-conducting porous electrolyte-filled media or membrane, which is sandwiched between and in contact with the two active, solid electrodes. Its roles are to prevent direct electronic contact between the two electrodes that would result in

a short-circuit, to allow the flow of ionic species within the cell and certain separator materials can function as an internal cell safety device. The separator affects the internal cell resistance, stability, cycle-life, operating temperature range and cell kinetic parameters such as discharge and charge rates. By its ability to regulate the electrolyte distribution between the active electrodes, as a result of its morphology and permeability characteristics, the separator limits ionic diffusion and recombination rates, and, thus, impacts the cell capacity, power and available energy. With respect to the inherent safety of a lithium-ion cell, the separator may afford passive protection and tolerance to cell abuse conditions that may arise, and it can enhance the overall cell safety by providing another level of internal redundancy.

Battery Separators

The structural and physiochemical properties of the separator material strongly influence the overall cell performance, although the separator does not “actively” participate in the battery operation. The mere presence of the separator component within a cell adversely affects the overall cell performance, as it adds electrical resistance and occupies limited space. For electrochemical cell chemistries in which the liquid electrolyte solution serves only as an ionic conductor, the separator should be as thin as possible and porous, but yet possesses the required physical strength to maintain the mechanical and electrical separation between the solid electrodes. These characteristics are essential in order to achieve high energy and power densities for applications requiring such performance attributes. Thus, the practical design of a separator is an art, as there is a compromise between the degree of porosity of the material and its mechanical strength. For battery safety, a state-of-the-art battery separator for utilization with lithium-based cell chemistries should have the inherent ability to shut the battery down if overheating occurs. Several comprehensive reviews of battery separators in general (Refs. 2 and 3) have appeared in the literature, as well as some with a primary emphasis on lithium-based cell technologies (Refs. 4 and 5).

Primary Separator Properties and Characteristics for Rechargeable Lithium-Based Cells

A minimal separator *thickness* is required for high energy and high power applications; however, this can have a negative impact on mechanical strength and on safety. Presently, commercial separators for lithium-ion cells are nominally $<30\text{ }\mu\text{m}$ in thickness.

For longevity, the cell separator must have a high degree of *chemical stability* to resist degradation and/or loss of mechanical strength due to reactivity with or dissolution by the electrolyte. Also, it must have *electrochemical stability* to the highly oxidative or reductive environments posed by the active electrode cell components. Coupled with analyses of the separator material, cycle life and calendar life testing data can yield qualitative and quantitative measures of the material stability; both conditions can degrade the separator material properties, and such can be reflected by an observed degradation in cell performance.

Optimal separator *porosity*, which is the ratio of void volume to apparent geometric volume, is critical for optimal performance and safety, as sufficient liquid electrolyte must be absorbed and retained within the pores in order to maintain a high level of ionic conductivity. Porosity can be measured using liquid or gas absorption methods. Typical commercial separators for lithium-ion cells have a porosity of ~ 40 percent. A uniform *pore size distribution* is desirable within the total area of a separator film in order to achieve a uniform current distribution within a cell. In addition, small, submicron *pore size* dimensions have proven effective in reducing the possibility of an internal short circuit by preventing the penetration of larger active electrode material particles or by dendritic lithium metal. Pore structure and distribution can be characterized by modern analytical porosimetry techniques (Ref. 6) and/or scanning electron microscopy (SEM).

The separator should also possess characteristics for a high degree of *wettability* in the electrolyte.

A critical functional separator property is its *permeability*, as the presence of a solid separator material significantly increases the effective resistance of the ion-conducting electrolyte. Permeability is a measure of the ability of a porous material to transmit a fluid, i.e., the liquid electrolyte solution in a lithium-ion cell. The permeability is related to the material's porosity and *tortuosity*, (Ref. 7) which is the ratio of mean effective capillary length to the separator thickness. Although an air permeability method could be used to characterize the separator permeability, *electrical resistance* (Ref. 3) or modern ac impedance measurements are more comprehensive for the separator in an actual electrochemical cell environment. Data from such measurements are useful performance indicators, as they describe a voltage loss within the cell during discharge and afford a measure of rate capabilities. The air permeability method is most useful for quality control purposes where rapid determination of lot-to-lot variability is the principal concern.

The overall mechanical strength of the separator is characterized in terms of several factors, with each possessing desired requirements and performance metrics. Tensile strength and properties are strongly dependent upon the material manufacturing process. Uniaxially-oriented separator films are generally preferred for material dimensional stability, as they exhibit a minimal tendency to shrink at elevated temperatures or when exposed to electrolyte. High puncture strength, which can be measured with a tensile tester, is important to prevent the penetration of particulate electrode material through the separator, especially during cell assembly, which could lead to an electrical short circuit. High mechanical strength is important, especially in a uniaxially-oriented separator film's machine direction, as the material is subjected to considerable tension in this direction during the winding operation of a spiral-wound battery fabrication procedure, and contraction of the width during the operation would be undesirable. Biaxially-oriented films are more uniformly strong in both the machine and transverse directions.

The chemical composition and structure/morphology attributes of the separator material, which significantly impact the optimization of the properties and characteristics summarized above, can be qualitatively and quantitatively assessed and characterized by a host of analytical spectroscopic, microscopic, thermal and mechanical methodologies that are available within a modern, state-of-the-art materials R&D infrastructure. Most of the separator properties described above can be measured or characterized by established American Society for Testing and Materials (ASTM) standard methods. Separator material manufactured for either specific aerospace-battery applications or for high-volume terrestrial markets should also be defect-free, should have uniform properties and should be capable of forming a good interface with the cell electrode components. Separator thermal properties and related characteristics will be discussed in a subsequent section of this review.

General Types of Separator Materials for Rechargeable Lithium-Based Cells

Generally, battery separators for lithium-based cells can be classified into three major types, with respect to their structure, composition and related properties (1) nonwoven fiber mats, (2) microporous polymeric membranes, and (3) inorganic composite membranes (Ref. 5). In most commercial liquid-electrolyte lithium-based cells, microporous membrane separators are employed, and this class will be the subject of a subsequent section.

Nonwoven separator mats, which are prepared by bonding either natural or synthetic fibers together by various methods, possess the attributes of high porosity and large pore sizes, but thin structures generally lack the physical properties required for utilization in lithium-based cells. Such porous structures can usefully serve as mechanical support structures for gel polymer electrolytes, which can be impregnated into the fibrous mat (Ref. 8). A gel polymer electrolyte cell component can be inherently safer than a flammable organic liquid electrolyte, as

a result of decreased volatility. Depending upon the specific chemistry, a gel polymer electrolyte could possess sufficient mechanical integrity on its own to serve as both electrolyte and separator, such as was the case for the “Bellcore” cell technology (Ref. 9) of the mid-1990’s.

An inorganic composite or ceramic membrane, formed by bonding highly hydrophilic/wettable, nano-sized inorganic particles such as alumina, silica or zirconia together would possess the additional desirable properties of exceptional thermal stability and dimensional stability. However, it would be difficult for a thin film of such material to have the mechanical integrity necessary for cell winding and assembly. This may not be as significant of an issue for a prismatic cell design. Recently, Evonik Degussa Corporation has introduced a series of Separion separator materials into the marketplace which combine the desirable properties of both ceramic and polymeric nonwoven materials (Ref. 10). Possessing both excellent cell performance and thermal safety and abuse characteristics, this separator material is currently being developed further for hybrid electric vehicle applications (Ref. 11).

Microporous Polymeric Membranes

Presently, most commercially-available lithium-ion spiral-wound cells containing a liquid organic electrolyte employ a microporous polymeric membrane separator due to advantages of performance, safety and cost (Refs. 3 and 5). These separators are based on polyolefin materials, such as polyethylene (PE), polypropylene (PP) and blends of such. Such polyolefin-based separators, which afford both excellent chemical stability and mechanical properties, are manufactured by either a wet or a dry process, both of which employ orientation steps to impart porosity and increase tensile strength.

The dry process involves no use of solvents, which could be a source of separator material contamination, and involves (1) melting a polyolefin resin, (2) extruding the resin into a film, (3) thermal annealing, and (4) stretching under controlled thermal conditions to create desired structural micropores (Ref. 4). Commercial polyethylene and polypropylene films prepared by this process are available from Celgard, LLC (Ref. 3). Celgard 2730 and Celgard 2400 are single layer PE and PP separators, respectively, while Celgard 2320 and 2325 are PP/PE/PP trilayer separator materials. As will be discussed in a subsequent section, the trilayer material structure can function in a lithium-ion cell as a “thermal fuse” for enhanced inherent cell safety, as well as providing exceptional puncture strength. The Celgard microporous separator materials have been extensively characterized as a lithium-ion cell component. Single-layer materials made from a blend of PE and PP have also been patented to afford enhanced cell safety characteristics (Ref. 12). Upon heating this blended material, which contains microporous regions of PE and PP, the separator impedance increases near the melting point of the PE and remains high until beyond the melting point of PP.

The wet process, or phase inversion process, (Refs. 13 and 14) involves mixing a polyolefin resin with a hydrocarbon liquid or low molecular oil, melting the mixture, extruding the melt into a sheet, orientating the sheet and extracting the liquid phase. Polyolefin battery separators made by the wet process are available from several sources, such as a widely used polyethylene-based material from ExxonMobil Chemical and its Japanese affiliate Tonen Chemical.

In Figure 1, the microstructures of microporous polyolefin separators made by the dry process (a) and the wet process (b) are compared. In general, separators made by the dry process exhibit a high orientation-dependent tensile strength, while membranes made by the wet process are nonoriented with respect to both pore structure and mechanical strength. A dry-process produced separator appears to be more suited for a high power density lithium-ion cell due to its open and straight porous structure, while the tortuous and interconnected porous structure of a wet-process made material is more suitable for a cell with a long cycle life requirement (Ref. 5).

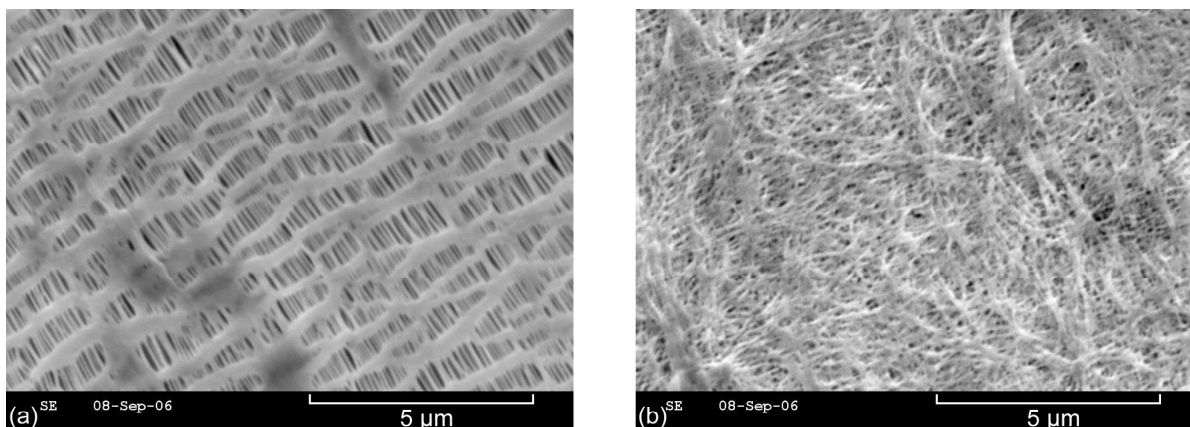


Figure 1.—Scanning electron micrographs of (a) a Celgard 2500 PP membrane made by the dry process and (b) a Tonen membrane made by the wet process. Separator samples were sputter-coated with gold to provide better resolution by reducing electrostatic charging (Ref. 19).

For practical lithium-ion cell performance most microporous separators have a thickness below 50 μm and average pore diameters below 1 μm . For high energy density applications, separator thicknesses of <25 μm are desirable in order to achieve a lower internal resistance, however, the safety concern with respect to mechanical integrity and penetration must be considered.

Lithium-ion Cell Performance and Safety: Separator Effects

Although classified as an inactive cell component, the separator properties play a critical role in obtaining practical and optimal cell-level performance and the inherent safety of the cell. In addition to initially possessing optimal properties for meeting the requirements for a specific application, the material properties must be uniform and stable over the desired application/mission life. The topic of separator performance degradation will be addressed in a subsequent section. With respect to cell and battery-level safety, the internal cell separator can complement safety control circuits possessing redundant safety features (e.g., CID, PTC, vent, etc.).

As mentioned earlier, the separator should possess a uniform pore structure and exhibit desired dimensional stability (e.g., low shrinkage) and a low electrical resistance in order to achieve good performance. The separator property of a low electrical resistance, which is a function of multiple material properties, has a pronounced effect upon such cell properties as capacity, rate performance, fast charge capability, overall cell resistance and cycle life. For safety concerns, the advantages of utilizing a thin separator must be weighed against the reduction in mechanical integrity, and the cell design should reflect such. External mechanical abuse (e.g., “nail” penetration or crush) or an internal short circuit could result in an abnormal increase in cell temperature, which, as discussed below, could seriously compromise safety. If an undesirable cell overcharge condition occurs, dendritic lithium metal could form, which could penetrate the separator as well as causing a drastic reduction in thermal stability.

Thermal Properties and Separator Shutdown Performance

If a lithium-ion cell is accidentally overcharged or abused, heat can be generated that could seriously compromise cell and battery-level safety, which is especially critical for manned, human-rated applications. Above a threshold temperature, a “self-heating” condition could occur due to exothermic reactions occurring internally within the cell (Ref. 15). Such reactions may include reactions between lithium and electrolyte and the thermal decomposition of internal cell

components. If the internal heat generation is allowed to continue, a catastrophic cell “thermal runaway” condition could occur, which would be a serious safety concern for a manned application.

The *shutdown* property of a polymeric separator material can internally provide a margin of safety against an external short circuit, accidental overcharge or an abuse condition resulting in an elevated cell temperature. Near the melting temperature of the specific polymeric material, the pores collapse, and this is accompanied by a significant increase in the separator and overall cell impedances, resulting in a termination of electrochemical activity and current flow. The shutdown property of a separator material can be characterized by monitoring the electrolyte-saturated separator material impedance while the temperature is ramped (Ref. 16). Optimization of the shutdown response and efficiency may be achievable by tailoring material properties and processing methods, and by employing additives to remove impurities. The shutdown mechanism would provide very little protection from an internal short circuit accompanied by a rapid internal heating rate.

The separator material should also possess high-temperature *melt integrity* and exhibit mechanical robustness above the shutdown temperature. After a separator shutdown occurrence, the cell temperature is likely to continue to increase. The separator must maintain mechanical integrity and a high impedance as a function of time at elevated temperatures in order to prevent the electrodes from making physical contact and creating the safety hazard of an internal short circuit. Thermal mechanical analysis (TMA) can provide a measure of a material’s melt integrity.

Thermal shutdown separators have been fabricated for commercial lithium-ion cells, typically as multilayer polyolefin structures (Ref. 17). The Celgard trilayer PP/PE/PP separator material, for example, functions as such an inherent safety device due to the difference in the PE and PP melting points (~130 to 140, and ~165 °C, respectively) (Refs. 5 and 18). The inner PE layer is capable of melting and filling the pores to inhibit ionic conduction at a temperature lower than that associated with an uncontrollable thermal runaway, while the outer PP layers continue to provide mechanical strength up to its melting temperature. If a “nail” penetration or an accidental overcharge at a high current density were to occur, such a structure would provide little protection from rapid localized overheating. Figure 2 is a DSC plot of a Celgard 2325 trilayer separator, which shows the melting temperatures of the PE and PP component layers.

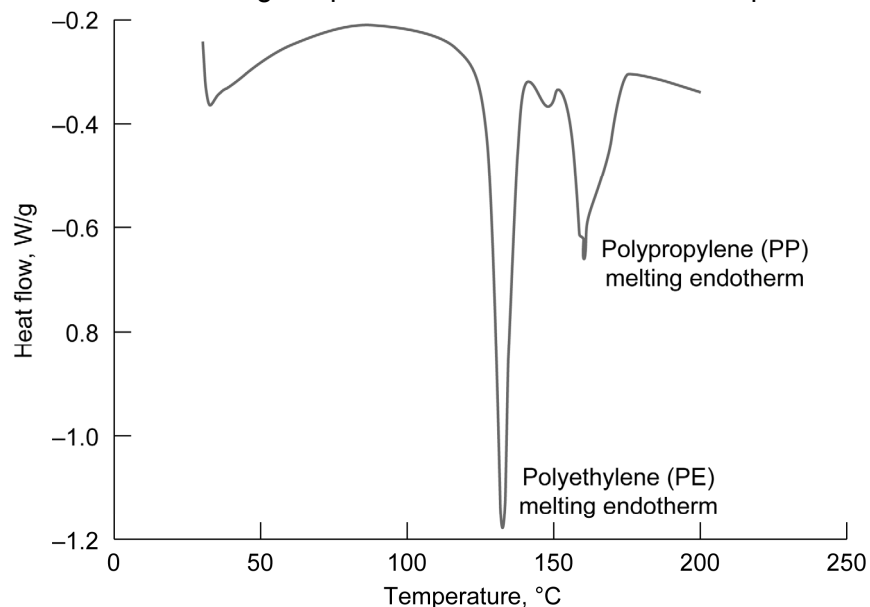


Figure 2.—Differential scanning calorimetry (DSC) data for a Celgard 2325 shutdown separator (Ref. 19).

With both U.S. Department of Energy's (DOE) and NASA Small Business Innovative Research (SBIR) program support, Policell Technologies, Inc. focused on the development of a novel separator material possessing a selectable thermal shutdown temperature which would be significantly lower than that of available commercial separator products in order to enhance large format, lithium-ion cell safety attributes (Ref. 20). The family of separator materials, which were engineered with respect to composition, could also be bonded via heat activation to the cell electrode surfaces for enhanced interfacial contact.

In support of the DOE Advanced Technology Development (ATD) Program for electric vehicle applications, Sandia National Laboratory (SNL) maintains an infrastructure dedicated to assessing and improving the thermal abuse tolerance of lithium-ion cells and cell components (Ref. 21). Employing a variety of experimental methodologies, responses of separator materials to abuse conditions are characterized and correlated with observed cell behavior (Ref. 22). Studies include shutdown performance, upper integrity temperature and separator breakdown leading to a cell thermal runaway condition.

Separator Degradation

Lithium-ion battery performance degradation and failure can often be attributed to the degradation of the separator component. Primary causes of separator degradation, excluding the previously-discussed thermal melting scenarios, are typically (Ref. 1) chemical attack by the electrolyte, (Ref. 2) electrochemical oxidation, (Ref. 3) dendritic growth of electrode material through the separator pores, (Ref. 4) structural degradation as a result of high temperature or cycling or (Ref. 5) clogging of separator pores during cycling. With respect to chemical stability, polyolefins exhibit excellent resistance to degradation in typical lithium-ion cell electrolytes.

During extended cycling, especially at elevated temperatures, the separator should be stable to the strongly oxidizing electrochemical environment on the surface facing the positive electrode and to the strongly reducing environment on the surface facing the negative electrode. Poor oxidation resistance can result in both poor high-temperature storage performance and long-term cycling behavior (Ref. 3). The superior oxidative resistance of polypropylene as compared to polyethylene results in a PP/PE/PP trilayer separator being more stable to an oxidative cell environment. Separator tolerance to an electrochemically oxidative environment will become more critical in the future, as a need for higher cell specific energies is addressed by employing higher positive voltage cathode materials. The superior mechanical integrity of the polypropylene outer layers of the trilayer material also affords puncture resistance.

When aged and/or cycled at elevated temperatures, lithium-ion cells may demonstrate a power loss that is partially attributed to a deterioration in separator performance (Ref. 23). It was shown that the cell impedance increased significantly, with a significant contribution being attributed to a loss in the ionic conductivity of the separator as a result of pores being blocked by electrolyte decomposition products.

Also in support of the DOE ATD Program for electric vehicle applications, extensive diagnostic studies were performed on aged, cycled and/or abused polyolefin separators in order to elucidate the causes and mechanisms of material degradation (Ref. 24). Dramatic changes in the separator surface morphology were observed; porosity losses, which were accompanied by increased separator resistance, were attributed to a clogging of the pores by solid deposits. The deposits resulted from electrolyte decomposition processes occurring at the electrode/electrolyte interfaces.

The high voltage stability of the separator material at an elevated temperature following a successful thermally-induced shutdown is an important requirement for series-connected cells (Ref. 22). A high-impedance cell in a series string could be subjected to high voltages from the

remaining cells, leading to a voltage breakdown of the separator caused by local currents through pinhole defects. Such a mechanism could result in localized heating and an internal short, which could ultimately result in thermal runaway and explosive cell failure.

Future Direction

Historically, only within the past decade have lithium-based cell separator materials been a strong focus of attention for enhanced development and optimization efforts, in order to address specific battery application performance requirements. As emphasized in this review, the separator component of advanced lithium-ion and lithium-based cells for high specific energy and/or high power density applications will play an important role in realizing optimal levels of performance and safety. It will be important to optimize all cell and battery/system-level components to enhance performance and reliability while maintaining and enhancing the safety of the technology. As lithium-based cell technology continues to become more advanced and mature, the requirements for and function of the separator will become more demanding and complex. Eventually, the goal of realizing an infinitely thin separator with negligible resistance will be met by a future generation of lithium-based cell technology that will practically employ polymeric or solid-state electrolytes.

As mentioned, energy storage needs and requirements for future NASA manned aerospace missions will demand high performance, high reliability and exceptional safety from battery technologies earmarked or selected for specific applications. The separator cell component will need to meet all of the key criteria necessary to be qualified for a particular battery type, and it will be critical that all components are assessed, characterized and qualified in appropriate multi-component and system-level configurations. Fortunately, at this point in time when NASA is investing resources in technology development for future human-rated aerospace missions, other governmental entities, coupled with the commercial sector, are also investing resources in similar technologies to address their unique future needs. Thus, viable opportunities for interagency collaborations and technology leveraging currently exist.

In similarity to addressing the technical challenges being addressed by NASA for achieving enhanced safety, reliability and performance of lithium-ion cells for human-rated mission applications, the U.S. Department of Energy has invested heavily in developing and maturing advanced electrochemical and battery technologies, particularly for future manned hybrid and all-electric vehicle applications. To meet future terrestrial consumer needs, it is highly recognized that “the cell separator component is the key to the safety of the battery” (Ref. 25).

To address the future technical performance and safety requirements that will be required for the advanced lithium-ion battery technologies that will eventually enhance and enable future NASA Exploration Mission and Constellation Program aerospace missions, the NASA Exploration Technology Development Program (ETDP) is currently supporting the Lithium-based Battery Development element of the Energy Storage Project (Ref. 26), which also includes an advanced proton-exchange membrane fuel cell development element. Lithium-ion cell component development subtasks, which are focused on achieving necessary cell-level performance and safety goals, are a critical part of this program. A focused separator development subtask is a key part of this NASA program, and it will address technology gaps between contemporary material performance and needs to meet future aerospace mission requirements. The programmatic approach for this subtask will emphasize the leveraging of current government and private-sector technology development activities.

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